IL-0148-19

Illinois Refining Division

Marathon Petroleum Company
Robinson, Illinois 62454
Telephone 618/544-2121

CERTIFIED MAIL - RETURN RECEIPT REQUESTED
CERTIFIED NO. P 670 874 821

February 21, 1985

Mr. Mark Haney
Illinois Environmental Protection Agency
2200 Churchill Road
Springfield, IL 62706

Dear Mr. Haney:

The fourth quarterly ground water sampling of 1984 was implemented with modifications as per your letter of November 19, 1984. Wells B-4 and N-2 were sampled on December 3, 1984. One sample bottle, TOX for well N-2, froze and broke in transit to the laboratory. The well was re-sampled for TOX on December 19, 1984.

Enclosed are copies of the contract laboratory's analysis results, along with QA/QC data. The pH and conductance, run by the refinery laboratory immediately after samples were taken, are included in these reports and labeled "(field)". The Fecal Coliform measurements done by the refinery lab were not included in these reports. Fecal Coliform was zero (0) for Well #B-4.

In my letter to you of January 2, 1985, the QA/QC program for the refinery laboratory for pH, specific conductance, and Fecal Coliform was discussed. This program is being implemented as follows:

a. Conductance and pH meters were calibrated with known standards immediately before the December 3, 1984 analyses were done. See Attachment I.

b. A set of unknowns for pH and conductivity has been received from Environmental Resource Associates and will be analyzed in late February/early March.

c. A set of unknowns including pH has been received from the USEPA and will be analyzed in late February/early March.

If you have any questions on the above, please call me at 618-544-2121.

Sincerely,

David R. Saad
Environmental Coordinator

DRS:lmw
Attachment

ATTACHMENT I

pH Meter - Calibrated with pH 4 standard
pH 7 standard

Conductance Meter - Calibrated and then tested standards

Standard	Value Obtained
40	41
600	588
3,000	2,469

RECEIVED
FEB 22 1985
IEPA-DLPC

Technical Report

for

MARATHON PETROLEUM COMPANY

MARATHON AVE.

ROBINSON, IL 62454

REVISED

ENVIRONMENTAL DEPT.

Chain of Custody Data Required for ETC Data Management Summary Reports

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
F9337	MARATHON PETROLEUM COMPANY	MPCROBGWM	WN2-D	841203	1330	

June D. Schaper for
Denis C. K. Lin, Ph.D.
Vice President
Research and Operations

TABLE OF CONTENTS

Introduction

This report contains the analytical results on your water sample, WN2-D 84/12/03 13:30. It is designed to include comprehensive data from the entire analytical process in order to satisfy the needs of various levels of review.

The results obtained from your sample are presented in tabular format immediately following this introduction. Quality assurance data is tabulated along with the appropriate sample results for verification. Depending on the analyses ordered, the quality assurance data may include results from blank, spiked blank, spiked sample (i.e. matrix spike) and replicate sample as well as results from surrogate compound analyses. Quality assurance data for verification of proper instrument performance is also included where appropriate. The report appendices include the chain of custody record for your sample and, where appropriate, the gas chromatograms and mass spectra.

The procedures used in the analysis of the sample are described in this report's methodology section. All analytical procedures within our laboratory are performed within a strictly enforced Quality Assurance Protocol. A description of this Protocol is included in the report.

Results

Sample results, and associated quality assurance data, are always tabulated in one or more of this report's Quantitative Results Tables. The format of each table varies with the class of analysis.

ETC ENVIRONMENTAL TESTING and CERTIFICATION

JAN 12, 1985

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA

Ground Water Monitoring - Conventional Analysis Data (QR10)

Chain of Custody Data Required for ETC Data Management Summary Reports

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
F9337	MARATHON PETROLEUM COMPANY	MPCROBGWM	WN2-D	841203	1330	

NPDES Number	Compound	Results									
		Sample Concen mg/l	MDL mg/l								
	Total Organic Carbon	35	1								
	Total Organic Carbon	36	1								
	Total Organic Carbon	36	1								
	Total Organic Carbon	36	1								

ETC ENVIRONMENTAL TESTING and CERTIFICATION

FEB 8, 1985

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA

Field Parameters (QR16)

Chain of Custody Data Required for ETC Data Management Summary Reports

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
F9337	MARATHON PETROLEUM COMPANY	MPCROBGMM	WN2-D	841203	1330	

Parameter		Results: Sample Measure	Results: MDL								
pH (Field)	std	6.56									
pH (Field)	std	6.55									
pH (Field)	std	6.54									
pH (Field)	std	6.54									
Specific Conductance (Field	um/cm	400									
Specific Conductance (Field	um/cm	400									
Specific Conductance (Field	um/cm	400									
Specific Conductance (Field	um/cm	400									
pH	std	8.20									
pH	std	8.20									
pH	std	8.20									
pH	std	8.20									
Specific Conductance	um/cm	510									
Specific Conductance	um/cm	560									
Specific Conductance	um/cm	510									
Specific Conductance	um/cm	500									

Appendix E

Chain-of Custody Forms

1) A field Chain-of-Custody form (CC1) is included for all samples shipped by ETC shuttle.

2) An in-house sample Chain-of Custody form is included for the period the sample was in ETC's possession.

3) A subcontractor's Chain-of-Custody form is included for any analytical work not performed within ETC's laboratory.

4) Any additional Chain-of-Custody material provided by a client or by a client's sampling agent is also included.

ETC TESTING and CERTIFICATION

CHAIN OF CUSTODY FORM (CC1)

Seal Date: 11/20/84
Sealed By: Herberg

SHIP TO:

Company: Marathon Petroleum Co
Facility/Site: Marathon Ave
Address: Robinson, IL 62454
Attn.: Vicky May
Phone: ( ) -

SAMPLE IDENTIFICATION

Facility/Site Code: MPCR0B6WM

Source Codes:

Well	(W)	River/Stream	(R)	Surface Impoundment	(I)	Lake/Ocean	(L)
Soil	(S)	Bottom Sediment	(B)	Pretreatment Facility	(P)	Treatment Facility	(T)
Outfall	(O)	Generation Point	(G)	Leachate Collection Sys.	(C)	Other	(X)

Source Code (from above)	Your Sample Point ID (left justify)	Start Date (mo/day/yr)	Start Time (2400 hr. clock)	Elapsed Hours (composite)
W	N2	11/28/84	1330	
Example: W	10/J2631P	05/15/81	0930	13

SHUTTLE CONTENTS

Sample Bottle	Condition	Sample Bottle	Condition
F9337 TX1	Frozen	Broken	
F9337 B1	Frozen		
F9337 A1	Frozen		

CHAIN OF CUSTODY CHRONICLE

1. Shuttle Opened By: (print) Vicki May Date: 11/28/84 Time: 3:45
Signature: Vicki May Seal #: 00338493 Intact: X

2. I have received these materials in good condition from the above person.
Name: Signature:
Date: Time: Remarks:

3. I have received these materials in good condition from the above person.
Name: Signature:
Date: Time: Remarks:

4. I have received these materials in good condition from the above person.
Name: Signature:
Date: Time: Remarks:

5. Shuttle Sealed By: (print) Date: Time:
Signature: Seal #:

ETC USE ONLY Opened By: Jacob Date: 12-4-84 Time: 1:30
Seal #: 33834 Condition: ok

ETC ENVIRONMENTAL TESTING and CERTIFICATION

F9337
ETC Sample No.:

SAMPLE POINT INFORMATION FORM (CC2)

FIELD MEASUREMENT DATA

Select up to 3 parameters to be recorded by entering the appropriate code letter in the first space for each of the 3 data entry fields provided. Enter the actual measurement data (in the units specified) for the three parameters you have selected.

PARAMETERS		ACTUAL (left justify)	EXAMPLE
Flow (CFS)	A		C 2.5
Volume (Gal)	B		
Sample Depth (Ft)	C		
Depth to Water (Ft)	D		F 5.0
Discharge Rate (GPM)	E		
Depth to Bottom (Ft)	F		
Event Time (2400-Hr Clock)	G		G 1103
Depth to Screen (Ft)	H		

FIELD TEST DATA

DO (Mg/L) [illegible]

Sample Temp. (°C)

	Single Measurement	2nd of Quadruplicate	3rd of Quadruplicate	4th of Quadruplicate
pH	[illegible]	6.55	6.57	
Specific Conductance (uMHOS/CM)	[illegible]	4,100	4,000	4,200

THE FOLLOWING DATA IS FOR YOUR RECORDS ONLY

SAMPLING METHOD (choose one)

AIR-LIFT PUMP ( )
AUGER.....(X)
BAILER.....( )
BOTTLE.....( )
COLIWASA.....( )
DIPPER.....( )
KEMMERER.....( )
MISKIN.....( )
PERISTALTIC PUMP....( )
PETERSEN.....( )
PISTON PUMP.....( )
SCOOP/SHOVEL.....( )
SQUEEZE PUMP.....( )
SUBMERSIBLE PUMP.( )
SUCTION LIFT PUMP ( )
SURGER.....( )
THIEF.....( )
TRIER.....( )
VEHMEYER..( )
OTHER _____

SAMPLE TYPE (choose one)

GRAB (X)
COMPOSITE ( )
OTHER ( )

(describe) (describe)

WEATHER
[illegible]

SAMPLE DESCRIPTION (e.g.,color,odor)
[illegible]

(describe) (describe)

Form Prepared By: Vicki May
name (print)

Employer: Marathon

From Page No.__

Log #	Sample #	TOC
7376	F9336	16.58
		16.58
		16.18
		16.49
	F9337	35.29
		36.09
		35.88
		35.65
	400 ppm	403.7
7373	F9985	6.590
		5.413
		6.015
		6.138
7414	F9220	2.226
		2.735
	F9222	6.407
		6.851
	F9223	2.064
		1.640
	F9224	1.962
		1.784
	400 ppm	408.3
	F9225	2.348
		1.325
	F9226	1.427
		1.245
	F9227	1.696
		1.570
	F9228	2.228
		1.691
	F9229	2.354
		1.619
	F9230	3.340
		2.867
	F9231	2.580
		3.364

Log #	Sample #	TOC
	400 ppm	407.4
7340	G1562	1.458
		1.356
		1.730
		1.528
	G1563	2.500
		2.408
		2.707
		2.647
	G1564	1.446
		1.583
		1.472
		1.887
	400 ppm	413.8
Low Spike	G1564-S	21.28
		21.77
High Spike	F9337-S	202.0
		202.3

To Page No.__

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by S. McArthur	12/29/84

Project No. _____
Book No. _____ TITLE PH/SCOND

From Page No. _____

Sample	PH	Temp	Kw	K	Range	Scond
~~G4840~~	7.8	10.5	1.023	1.42	2 mV	1400
	7.8	10.4	1.046	1.45	"	1500
	7.8	10.4	1.035	1.44	"	1400
	7.8	10.4	1.062	1.47	"	1500
G4841	7.4	12.5	0.976	1.28	"	1300
	7.4	12.6	0.951	1.30	"	↓
	7.4	12.6	0.964	1.32	"	
	7.4	12.6	0.971	1.33	"	
G4842	~~8.0~~ 7.8	12.4	1.078	1.42	"	1400
	7.7	12.4	0.980	1.29	"	1300
	7.7	12.4	0.987	1.30	"	↓
	7.7	12.4	0.993	1.31	"	
F9337	8.2	17.2	0.435	0.511	"	510
	8.2	17.2	0.475	0.558	"	560
	8.2	17.2	0.432	0.508	"	510
	8.2	17.2	0.429	0.504	"	500
G4222	8.0	17.4	0.478	0.559	"	560
	8.0	17.4	0.477	0.558	"	↓
	8.0	17.4	0.475	0.556	"	
	8.0	17.4	0.474	0.554	"	550

Calculations by [signature]

To Page N

Witnessed & Understood by me	Date	Invented by	Date
[signature] 1/26/05		Recorded by [signature]	

From Page No. ___

Sample	PH	Temp	Kw	K	Range	Scond
G4840	7.8	10.5	1.023	1.42	2 mV	1400
	7.8	10.4	1.046	1.45	"	1500
	7.8	10.4	1.035	1.44	"	1400
	7.8	10.4	1.062	1.47	"	1500
G4841	7.4	12.5	0.976	1.28	"	1300
	7.4	12.6	0.951	1.30	"	↓
	7.4	12.6	0.964	1.32	"	
	7.4	12.6	0.971	1.33	"	
G4842	~~8.0~~ 7.8	12.4	1.078	1.42	"	1400
	7.7	12.4	0.980	1.29	"	1300
	7.7	12.4	0.987	1.30	"	↓
	7.7	12.4	0.993	1.31	"	
E9337	8.2	17.2	0.435	0.511	"	510
	8.2	17.2	0.475	0.558	"	560
	8.2	17.2	0.432	0.508	"	510
	8.2	17.2	0.429	0.504	"	500
G4222	8.0	17.4	0.478	0.559	"	560
	8.0	17.4	0.477	0.558	"	↓
	8.0	17.4	0.475	0.556	"	
	8.0	17.4	0.474	0.554	"	550

Calculations by [signature]

To Page

Witnessed & Understood by me	Date	Invented by	Date
[signature] 1/26/05		Recorded by [signature]	

Technical Report

for

MARATHON PETROLEUM COMPANY

MARATHON AVE.

ROBINSON, IL 62454

REVISED

Chain of Custody Data Required for ETC Data Management Summary Reports

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
F9336	MARATHON PETROLEUM COMPANY	MPCROBGWM	WB4-D	841203	0900	

June G. Schaper/for
Denis C. K. Lin, Ph.D.
Vice President
Research and Operations

ETC ENVIRONMENTAL TESTING and CERTIFICATION CORPORATION

DENIS C.K. LIN, Ph.D.
Vice President and
General Manager

February 9, 1985

Ms. Vicky May
Marathon Petroleum Company
Marathon Avenue
Robinson, IL 62454

Dear Mr. May:

Enclosed are corrected reports for your samples WB4-D 841203 0900 and WN2-D 841203 1330, (ETC samples F9336-F9337, respectively). We have added pH and Specific Conductance results from our lab which was originally omitted from these reports.

If you have any questions regarding your report, we encourage you to contact any one of the following persons in our Client Service organization:

Lynne Fanjoy	(201) 225-6747
Ellen Leinfuss	(201) 225-6715
Pat McIsaac	(201) 225-6751
Steven Timmerman	(201) 225-6780

They will coordinate your inquiries with the appropriate technical personnel. Your account executives, along with our Client Service organization are also available to assist you in defining requirements for future testing programs.

If we can be of further service to your organization in the future, please contact us.

Sincerely,

June D. Schaper /fa

Denis C. K. Lin, Ph.D.

DCKL:rp
Attachments

284 RARITAN CENTER PARKWAY • CN 3154 • EDISON, NJ 08818 • (201) 225-6707 225-6700

TABLE OF CONTENTS

Introduction

This report contains the analytical results on your water sample, WB4-D 84/12/03 09:00. It is designed to include comprehensive data from the entire analytical process in order to satisfy the needs of various levels of review.

The results obtained from your sample are presented in tabular format immediately following this introduction. Quality assurance data is tabulated along with the appropriate sample results for verification. Depending on the analyses ordered, the quality assurance data may include results from blank, spiked blank, spiked sample (i.e. matrix spike) and replicate sample as well as results from surrogate compound analyses. Quality assurance data for verification of proper instrument performance is also included where appropriate. The report appendices include the chain of custody record for your sample and, where appropriate, the gas chromatograms and mass spectra.

The procedures used in the analysis of the sample are described in this report's methodology section. All analytical procedures within our laboratory are performed within a strictly enforced Quality Assurance Protocol. A description of this Protocol is included in the report.

Results

Sample results, and associated quality assurance data, are always tabulated in one or more of this report's Quantitative Results Tables. The format of each table varies with the class of analysis.

ETC ENVIRONMENTAL TESTING and CERTIFICATION

JAN 6, 1985

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA

Ground Water Monitoring Pesticides and Herbicides (QR09)

Chain of Custody Data Required for ETC Data Management Summary Reports

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
F9336	MARATHON PETROLEUM COMPANY	MPCROBGMM	WB4-D	841203	0900	

Parameter	Results		QC Replicate		QC Blank and Spiked Blank			QC Matrix Spike		
	Sample Concen. ug/l	MDL ug/l *	First ug/l	Second ug/l	Blank Data ug/l	Concen. Added ug/l	% Recov	Unspiked Sample ug/l	Concen. Added ug/l	% Recov
Endrin (GC)	ND	.10	ND	ND	ND	0.2	77	ND	0.2	68
Lindane (GC)	ND	2	ND	ND	ND	4.0	92	ND	4.0	92
Methoxychlor (GC)	ND	50	ND	ND	ND	100	94	ND	100	88
Toxaphene (GC)	ND	2.50	ND	ND	ND	5.0	109	ND	5.0	74
2,4-D	ND	50	ND	ND	ND	100	69	ND	100	75
2,4,5-TP (Silvex)	ND	5	ND	ND	ND	10	165	ND	10	175

* GC/ECD MDL calculated for each sample matrix. < indicates compound was less than the sample MDL.

ETC ENVIRONMENTAL TESTING and CERTIFICATION

JAN 16, 1985

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA

Ground Water Monitoring Metals (QR41)

Chain of Custody Data Required for ETC Data Management Summary Reports

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
F9336	MARATHON PETROLEUM COMPANY	MPCROBGWM	WB4-D	841203	0900	

Compound	Results									
	Sample Concen. ug/l	MDL ug/l								
Arsenic	ND	5								
Barium	32	5								
Cadmium	ND	5								
Chromium	ND	20								
Lead	15	5								
Mercury	ND	.30								
Selenium	BMDL	5								
Silver	ND	10								
Iron	ND	200								
Manganese	1300	5								
Sodium	34000	1000								

ETC ENVIRONMENTAL TESTING and CERTIFICATION

JAN 12, 1985

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA

Ground Water Monitoring - Conventional Analysis Data (QR10)

Chain of Custody Data Required for ETC Data Management Summary Reports

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
F9336	MARATHON PETROLEUM COMPANY	MPCROBGWM	WB4-D	841203	0900	

NPDES Number	Compound		Results: Sample Concen.	Results: MDL
	Chloride	mg/l	2.00	.20
	Fluoride	mg/l	.34	.10
	Nitrate as N	mg/l	<.10	.10
	Sulfate as S04	mg/l	<2.00	2
	Phenolics, Total	mg/l	<.05	.05
	Total Organic Halides (TOX)	ug/l	6.80	5
	Total Organic Halides (TOX)	ug/l	11	5
	Total Organic Halides (TOX)	ug/l	11	5
	Total Organic Halides (TOX)	ug/l	8.70	5
	Total Organic Carbon	mg/l	17	1
	Total Organic Carbon	mg/l	17	1
	Total Organic Carbon	mg/l	16	1
	Total Organic Carbon	mg/l	16	1
	Gross Alpha	pCi/l	<1.50	1.50
	Gross Beta	pCi/l	5.30	1.20
	Gross Beta	pCi/l	+/-1.20	

ETC ENVIRONMENTAL TESTING and CERTIFICATION

FEB 8, 1985

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA

Field Parameters (QR16)

Chain of Custody Data Required for ETC Data Management Summary Reports

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
F9336	MARATHON PETROLEUM COMPANY	MPCROBGWM	WB4-D	841203	0900	

Parameter		Results									
		Sample Measure	MDL								
pH (Field)	std	7.15									
pH (Field)	std	7.19									
pH (Field)	std	7.20									
pH (Field)	std	7.20									
Specific Conductance (Field	um/cm	435									
Specific Conductance (Field	um/cm	426									
Specific Conductance (Field	um/cm	426									
Specific Conductance (Field	um/cm	426									
pH	std	7.80									
pH	std	7.80									
pH	std	7.80									
pH	std	7.80									
Specific Conductance	um/cm	500									
Specific Conductance	um/cm	500									
Specific Conductance	um/cm	500									
Specific Conductance	um/cm	500									

Methodology
for
GC Analysis of Herbicides and Pesticides

The methods employed in the analysis of your sample for herbicides and pesticides are established EPA methods taken from the "Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples," June, 1980.

The herbicide method can be summarized as follows: A measured volume of sample, approximately 500-1000 ml, to which sodium sulfate has been added, is acidified and extracted with methylene chloride. The methylene chloride extract is evaporated to dryness, and the residue is derivatized with diazomethane and injected into a gas chromatograph equipped with a ^{63}Ni electron capture detector.

The pesticide method can be summarized as follows: A measured volume of sample, approximately 500ml, is extracted with methylene chloride. The extract is dried and concentrated to a final volume of 1ml and injected into a gas chromatograph equipped with a ^{63}Ni electron capture detector.

The GC operating parameters were as follows:

COLUMN

6' x 4 mm glass 1.5% SP-2250 & 1.95% SP-2401
Supelcoport 100/120 mesh

CARRIER FLOW

60 ml/min. Argon/Methane

COLUMN OVEN

220° C

INJECTOR TEMPERATURE

225° C

DETECTOR TEMPERATURE

325° C

Methodology for Analysis of Metals

AQUECUS

The determination of metals in aqueous samples is performed according to the methods published by EPA in "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March, 1979, and Appendix IV of the Federal Register, December 3, 1979. Arsenic, selenium and thallium are determined by furnace AA; silver, aluminum, barium, beryllium, boron, cadmium, calcium, chromium, copper, cobalt, iron, magnesium, manganese, molybdenum, nickel, lead, sodium, antimony, tin, titanium, vanadium, and zinc are determined by ICP emission spectrometry, except where lower levels of detection are required: in these cases (e.g. lead in groundwater monitoring samples) furnace AA is used. All furnace AA parameters are run by method of standard additions. The determination of mercury is performed by cold vapor AA.

EP TOXICITY

The determination of metals in aqueous EP Toxicity leachates is performed according to the methods published by EPA in "Test Methods for Evaluating Solid Waste" EPA SW-846, July 1982, and Appendix IV of the Federal Register, Dec. 3, 1979. Silver, arsenic, barium, cadmium, chromium, lead and selenium are determined by ICP emission spectrometry. Mercury is determined using cold vapor AA. For leachates that are organic in nature, the analyses are performed according to the methods described under ***OIL/SLUDGE*** below.

SOIL/SEDIMENT

The determination of silver, beryllium, cadmium, chromium, copper, nickel, antimony, lead, and zinc in sediment samples is performed according to methods published by EPA in "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue", EPA 600/4-81-055, October 1980. Mercury is determined according to the sediment method published by EPA in "Method for Chemical Analysis of Water and Wastes", EPA 600/4-79-020, March 1979. Arsenic, selenium and thallium are determined by furnace AA using nitric acid in a closed decomposition vessel for sample digestion.

OIL/SLUDGE

The determination of silver, aluminum, boron, barium, beryllium, calcium, cadmium, copper, chromium, cobalt, iron, magnesium, manganese, molybdenum, sodium, nickel, lead, antimony, tin, titanium, vanadium, and zinc in sludge/petroleum-based samples is performed by ICP emission spectrometry using a magnesium nitrate dry ashing digestion technique. Arsenic, selenium and thallium are determined by furnace AA using nitric acid in a closed decomposition vessel for sample digestion. Mercury is determined by cold vapor AA using the same digestion technique.

HEXAVALENT CHROMIUM

The determination of dissolved hexavalent chromium in drinking and surface waters is performed according to the methods published by EPA in "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1983. For domestic and industrial wastes, Method 7195 in "Test Methods for Evaluating Solid Waste," SW-846, USEPA 1982 may also be employed depending upon the matrix and nature of interfering species

Methodology
for
Analysis of Total Organic Halides

The method employed in the analysis of your sample for Total Organic Halide (TOX) is the established EPA Method 450.1, November, 1980. This is identical to EPA Method 8.66 in SW-846, 2nd Ed., 7/82 for evaluation of TOX in drinking and ground waters as provided under 40 CFR 265.92.

The EPA TOX method, in summary, can be described as follows: A sample of water that has been protected against the loss of volatiles by the elimination of headspace in the sampling container, and is free of undissolved solids, is passed through a column containing 40 mg of activated carbon. The column is washed to remove any trapped inorganic halides, and is then pyrolized to convert the absorbed organohalides to a titratable species which is then measured by a microcoulometric detector.

ETC ENVIRONMENTAL TESTING and CERTIFICATION

Quality Assurance/Quality Control Procedures (QA/QC)

ETC bases its quality assurance protocols on the following government guidelines:

- "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA-600/4-79-019, March 1979;
- National Enforcement Investigation Center Policies, and Procedures manual; EPA-330/9-79-001-R, October 1979;
- the recommended guidelines for EPA Methods 624 and 625. (Federal Register, December 3, 1979, pp. 69532-69559);
- "Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples," EPA 600/8-80-038, June 1980; and
- "Determination of 2,3,7,8-TCDD in Soil and Sediment" EPA, Region VII, Kansas City, September 1983.

However we have modified our protocols to provide a higher level of QA/QC than the guidelines require. For example, we analyze a higher than required number of quality control samples and we pay especially careful attention to the certification of the "reference standard" compounds we use in analysis. Below are listed the key QA/QC elements for the methods we used.

Analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry

- Each batch of 13 samples consists of 9 customer samples (at a maximum), one blank sample, one spiked blank, one spiked sample and one replicate sample. This amounts to a 30% quality control factor.
- Three surrogate compounds are added to each sample in the batch of 13.
- A blind quality control sample is introduced to the laboratory for analysis on a weekly basis.
- Each GC/MS is checked and retuned, if necessary, at the beginning of each day to ensure that its performance on bromofluorobenzene (BFB) meets the EPA criteria.
- A calibration curve for quantitation is prepared using a mixture of Volatile Organic Priority Pollutant "standards" at a minimum of 3 different concentrations and using a mixture of 3 internal standards at a constant concentration.
- The calibration curve is verified with a mixture of priority pollutant standards every day. If the response factors factors vary greater than 10%, the instrument must be recalibrated.

Analysis of Organic Compounds Extracted in Acid or Base/Neutral Solutions by Gas Chromatography/Mass Spectrometry

- Each batch of 20 samples consists of 16 customer samples (at a maximum), one blank sample, one spiked blank (for water matrices), one sample spiked with the priority pollutant standard mixture and a duplicate customer sample. This amounts to a 20% quality control factor.
- Three surrogate compounds are added to each sample in the batch for Base/Neutral analysis
- Two surrogate compounds are added to each sample in the batch for Acid analysis
- A blind quality control sample is introduced to the laboratory for analysis on a weekly basis.

rev. 7/84

- Each GC/MS is checked and retuned, if necessary, at the beginning of each day to ensure that its performance on decafluorotriphenylphosphine (DFTPP) meets the EPA criteria.

- A calibration curve for quantitation is prepared using a mixture of standards composed of either the Organic Acid or Base Neutral Extractable Compounds at a minimum of 5 concentrations and using 2,2'-difluorobiphenyl as an internal standard.

Analysis of Metals

All Samples

- New standards are prepared for each batch of samples.

- Normal calibration is performed using a blank sample and four standards that have been through the sample preparation procedure. A regression analysis is used to construct the calibration curve.

- All EP Toxicity samples and all samples determined by furnace atomic absorption are calculated by the "method of additions".

- For each sample analysis that requires the use of the "method of additions" technique, a three point calibration is performed using U.S. EPA "Methods for Chemical Analysis of Water and Wastes, 1979". Results are obtained using linear regression analysis. Any regression with a coefficient of correlation below 0.990 is considered suspect, necessitating review of calibration data or sample re-analysis.

- In constructing the normal calibration curves the lowest concentration levels we use are values greater than or equal to 5 times the Instrumental Detection Limit (IDL).

- All calibration standards are analyzed in duplicate, at a minimum.

- Independent reference standards are used to check the accuracy of calibration standards.

- A check standard is analyzed every ten samples to validate the normal calibration curve.

- One customer sample out of every ten is analyzed in triplicate.

Homogeneous Samples (except for Mercury analysis)

Samples are analyzed in batches of 30 or less. For batches in which the sample matrices are homogeneous, the QC program is a minimum of 25% and consists of analyzing:

- 3 sets of triplicate analyses;

- 2 Replicate spikes;

- 1 independent reference standard;

- 4 Calibration standards (processed using the sample preparation method);

- 4 Calibration standards (without sample preparation); and

- 1 Reagent Blank.

Heterogeneous Samples (except for Mercury analysis)

Samples are analyzed in batches of 30 or less. For batches in which the sample matrices are heterogeneous, the QC program is a minimum of 35% and consists of analyzing:

- 3 sets of triplicate analyses;
- 2 Replicate spikes;
- 1 Replicate independent reference standards;
- 4 Calibration standards (processed using the sample preparation method);
- 1 Procedural Blank;
- 4 Calibration standards (without sample preparation); and
- 1 Reagent Blank.

Analysis of Mercury

To analyze samples for mercury we group them by matrix in batches of 20 or less. Our QC program is a minimum of 30% and consists of analyzing:

- each of the 30 customer samples in duplicate;
- 3 sets of triplicate anaylses;
- 2 Replicate spikes;
- 2 Replicate independent reference standards;
- 10 Calibration standards (processed using the sample preparation method); and
- 2 Procedural Blanks.

Analysis of Pesticides, Herbicides and PCB's by Gas Chromatography

Pesticide, herbicide and PCB samples are grouped in batches of 16 customer samples or less according to the type of analysis to be performed. The QC program for each of these three types of analyses is a minimum of 20% and consists of analyzing:

- 1 procedural blank sample(a reagent blank is analyzed in the case of non-water matrices);
- 1 spiked blank sample (the spiked blank is eliminated in the case of non-water matrices);
- 1 replicate sample;
- 1 replicate spiked sample; and
- 1 known refernece QC sample for at least each 100 samples analyzed.

The instrument is calibrated each run with three standards, and checked every 10 samples.

Analysis of Cyanides, Phenols, Fluoride, Chloride, Nitrate and Nitrite

- All parameters are analyzed using a Technicon Autoanalyzer II GT.
- 3 calibration standards are analyzed at the beginning and end of each batch.
- Each batch (up to 80 samples) consists of analyzing one blank, one spiked blank, one duplicate and spiked sample every 20 samples, and an EPA known reference sample.

Analysis of Total Organic Carbon (TOC)

TOC samples are analyzed on a daily basis with the number of samples analyzed per day dependent on the request for duplicate or quadruplicate analyses. The quality control program is designed to maintain the appropriate amount of QC and consists of the following elements:

- Daily instrument calibration
- One blank
- Standard recalibration every 10 samples
- Spiked samples at a low and high level
- Every sample is run in duplicate at a minimum

Analysis of Total Organic Halide (TOX)

- Blank reagent water for absolute carbon backround must contain less than 5 ug/l of halide (as chloride).
- Using a trichlorophenol standard, the mean adsorption efficiency must be within +/- 15% of the standard value.
- Calibration standards are run every 10 samples.
- Every sample is run in duplicate at a minimum.

Analysis of 2,3,7,8-TCDD (Dioxin) by GC/MS (SIM)

- Each sample is dosed with a known quantity of $^{13}C_{12}$-2,3,7,8-TCDD as internal standard and $^{37}Cl_4$-TCDD as surogate standard. The action limits for surrogate standard results is +/- 40% of the true value. Samples showing surrogate standard results outside of these limits are reextracted and reanalyzed.
- Two laboratory "method blanks" are run along with each set of 24 or fewer samples. The method blank is also dosed with the internal standard and surrrogate standard.
- At least one per set of 24 samples is run in duplicate to determine intralaboratory precision.
- Qualitative Requirements. The following are met in order to confirm the presence of native 2,3,7,8-TCDD:

  a. Isomer specificity must be demonstrated initially and verified once per 8-hour work shift. The verification consists of injecting a mixture containing TCDD isomers which elute close to 2,3,7,8-TCDD. The 2,3,7,8-TCDD must be separated from interferring isomers, with no more than 25% valley relative to the 2,3,7,8-TCDD peak.

  b. The 320/322 ratio is within the range of 0.67 to 0.87.

  c. Ions 320, 322, and 257 are all present and maximize together the signal to mean noise ratio must be 2.5 to 1 or better for all 3 ions.

  d. The retention time is equal (within 3 seconds) the retention time for the isotopically labeled 2,3,7,8-TCDD.

  e. At least one of the positives can be confirmed by obtaining partial scan spectra from mass 150 to mass 350. The partial scan guidelines are as follows:

  . the 320/324 ratio should be 1.58 +/- 0.16

  . the 257/259 ratio should be 1.03 +/- 0.10

. the 194/196 ratio should be 1.54 +/- 0.15

- One sample is spiked with native 2,3,7,8-TCDD at a level of 1.0 PPB (for soil) for each set of 24 or fewer samples.
- In cases where no native 2,3,7,8-TCDD is detected, the actual detection limit is estimated and reported based on a signal to noise ratio of 2.5 to 1 at ions 320 and 322.
- For each sample, the internal standard is present with at least a 10 to 1 signal to noise ratio for both mass 332 and mass 334. Also, the internal standard 332/334 ratio must be within the range of 0.67 to 0.87.

Subcontractor QA/QC

Each subcontractoer is required to maintain an appropriate level of quality control. To insure this, each subcontractor is required to submit to ETC the quality control data for all analyses it performs. This data is kept on file at ETC. In general, the amount of quality control required is one duplicate sample with one spiked sample for every ten analyses.

Chain-of-Custody

The chain-of-custody procedure is part of our quality assurance protocol. We believe our chain-of-custody record fully complies with the legal requirements of federal, state and local government agencies and of the courts of law. The record covers:

- labeling of sample bottles, packing the Sample Shuttle and transferring the Shuttle under seal to the custody of a shipper;
- outgoing shipping manifests;
- the chain-of-custody form completed by the person(s) breaking the Shuttle seal, taking the sample, resealing the Shuttle and transferring custody to a shipper;
- incoming shipping manifests;
- breaking the Shuttle's reseal;
- storing each labeled sample bottle in a secured area;
- disposition of each sample to an analyst or technician; and
- the use of the sample in each bottle in a testing procedure appropriate to the intended purpose of the sample.

The record shows for each link in this process:

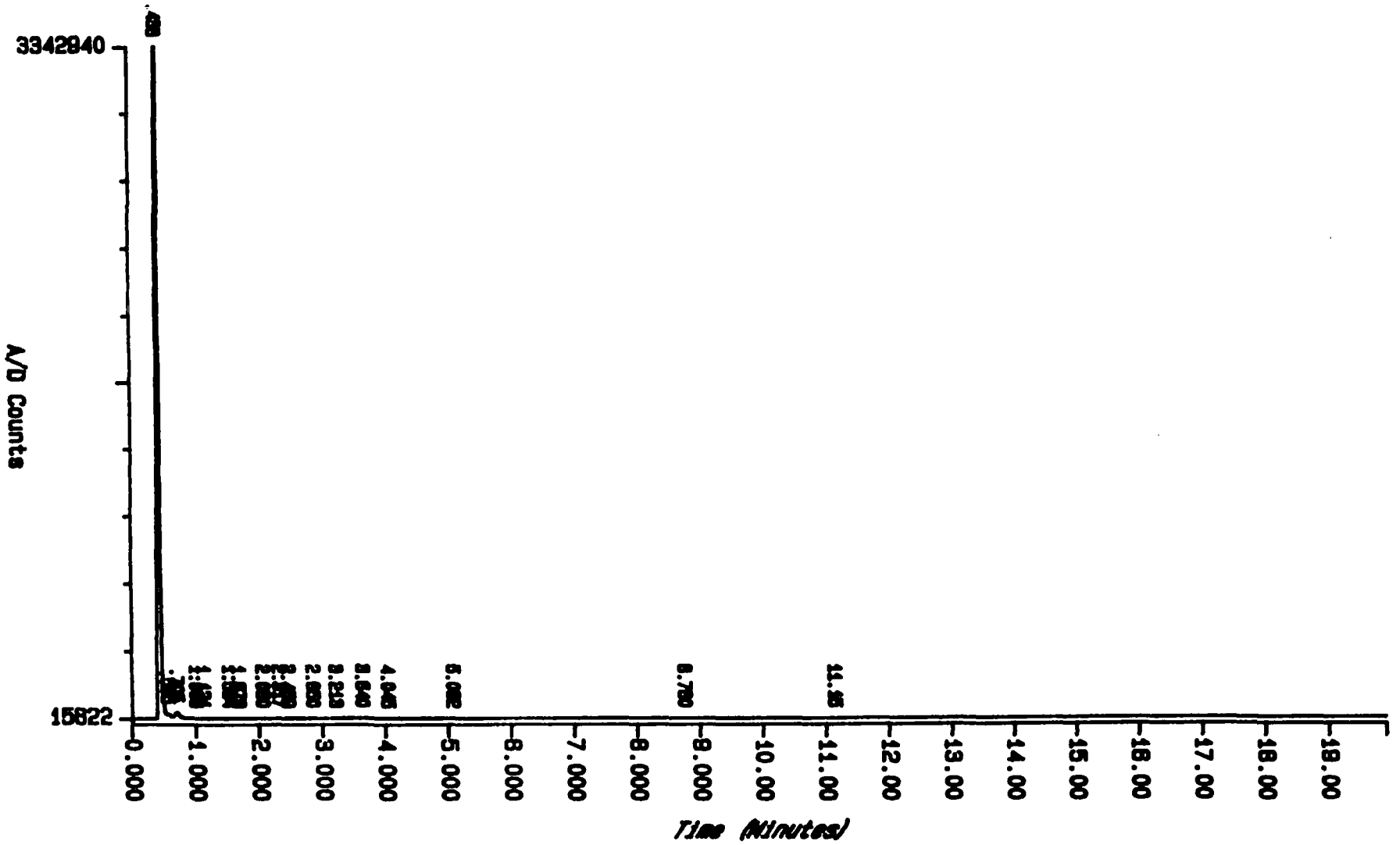
- the person with custody; and
- the time and date each person accepted or relinquished custody.

Appendix A1

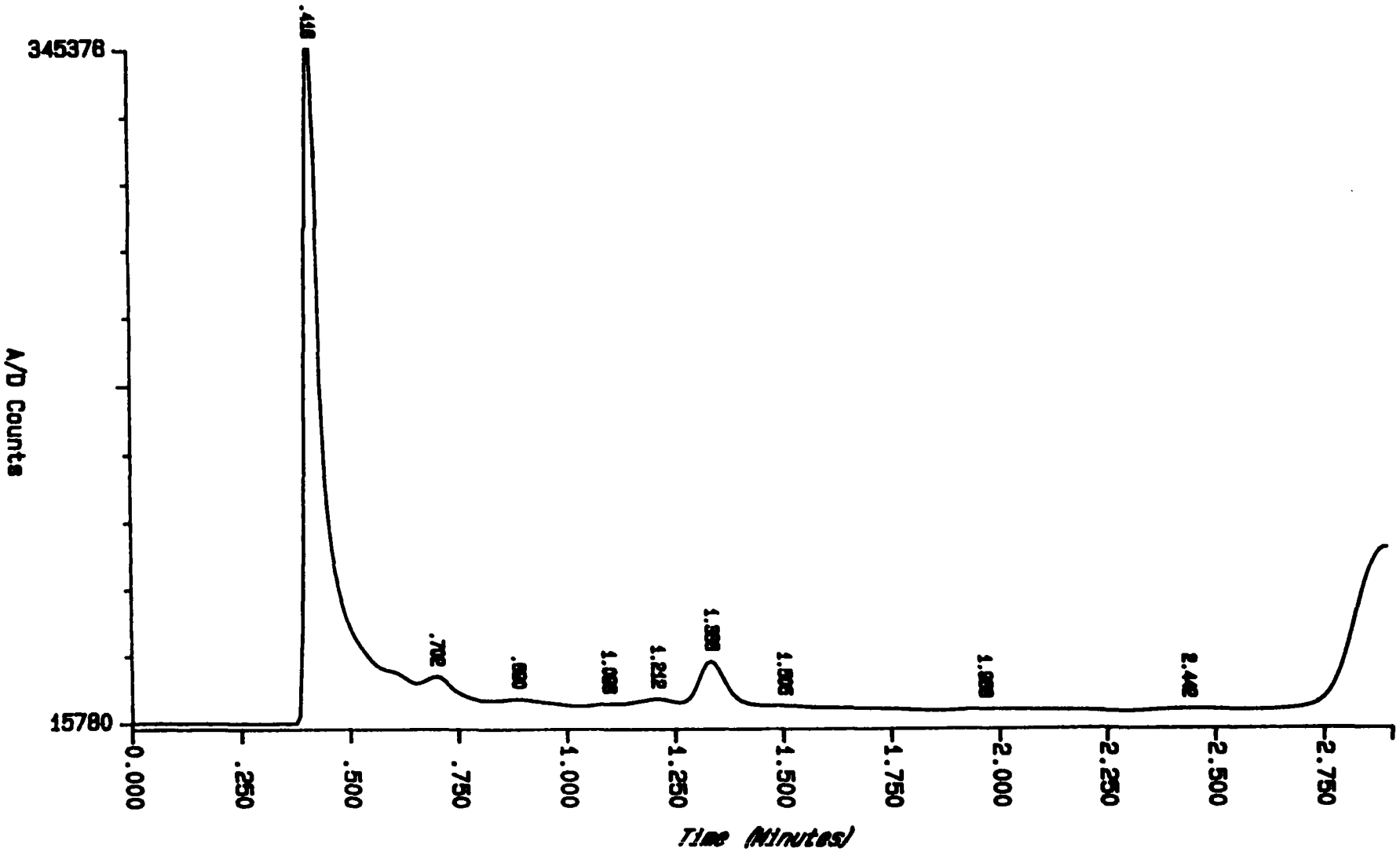
Gas Chromatographic Spectral Data for Quantitated Compounds

1) A reconstructed gas chromatogram for each sample analyzed by a GC instrument.

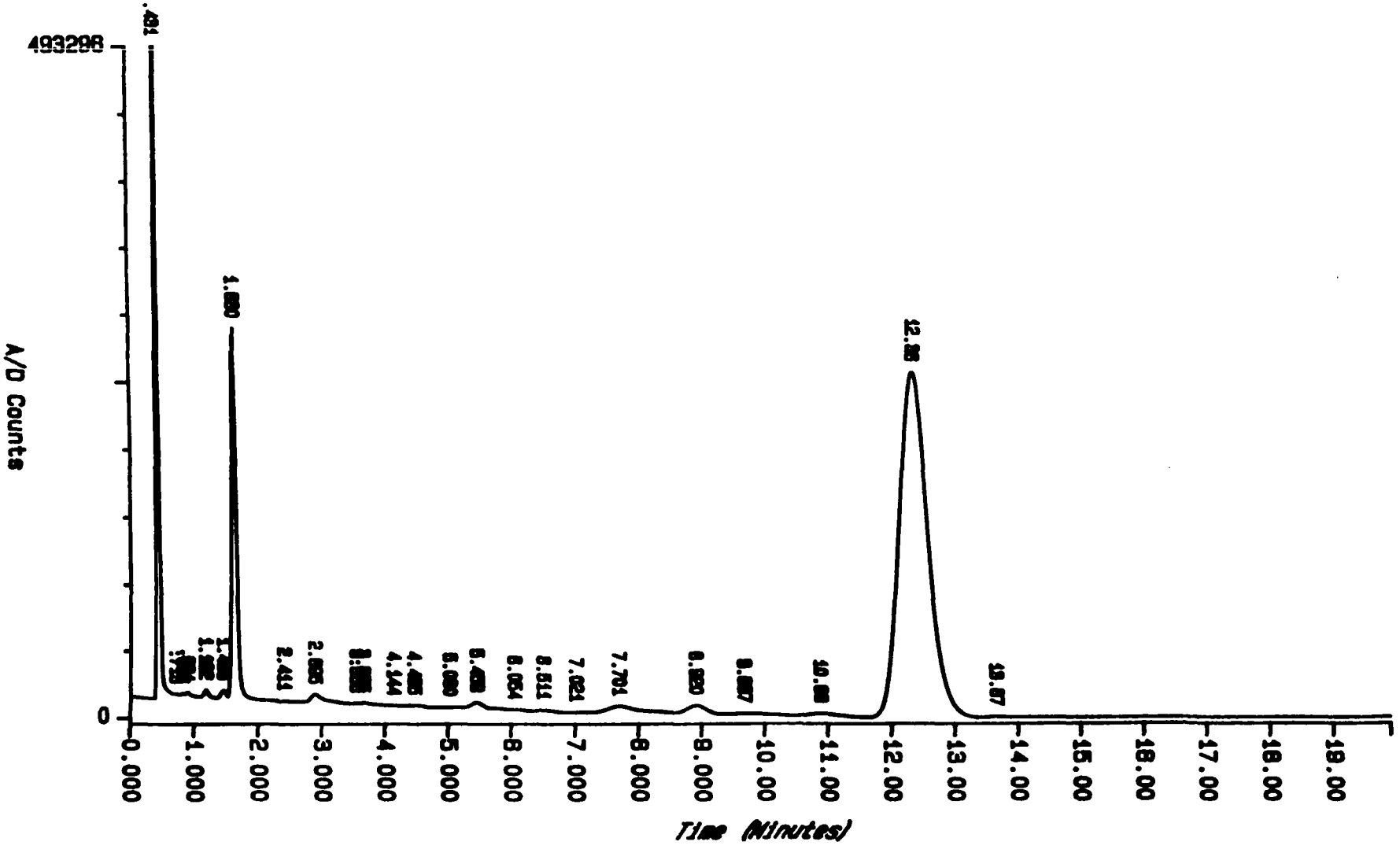
2) A reconstructed gas chromatogram for the appropriate standard compounds analyzed with the same GC under the same operating conditions.



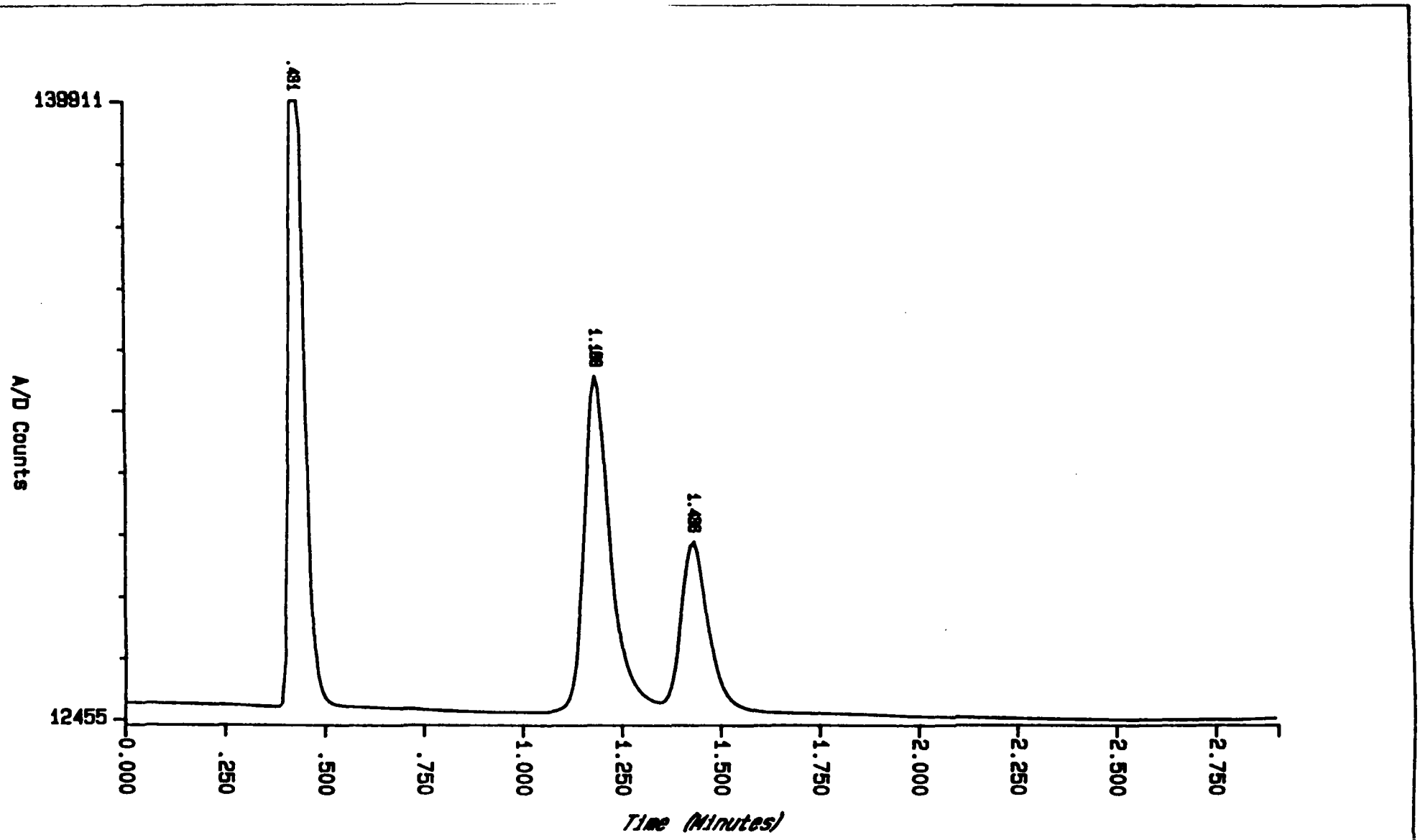
Sample: F9336 Injected at 22:24:11 ON DEC 27, 1984
Raw File: F9336D Proc File: F9336C Method: DWPST8



Sample: F8338 Injected at 15:28:12 ON DEC 27, 1884
Raw File: F8338B Proc File: F8338A Method: HERB8



Sample: DW STDB Injected at 18:10:25 ON DEC 27, 1984
Raw File: R9513 Proc File: P9513 Method: DWPST8



Sample: 24D 245TP A Injected at 15:06:30 ON DEC 27, 1984
Raw File: R9511 Proc File: P9511 Method: HERB8

Appendix D
Subcontractor's Data

1) A copy of the originating subcontractor's report is included for all data not generated within ETC's laboratory.

SDMS US EPA Region V

Imagery Insert Form

Some images in this document may be illegible or unavailable in SDMS. Please see reason(s) indicated below:

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[ ] **Other:**

ETC ENVIRONMENTAL TESTING AND CERTIFICATION

Subcontracted Analytical Results

ETC Job # F193361

Lab ID: L84830-C

Submitted by: MW CHYUN

Date: 12/20/84

Facility: [illegible] Sample Point: [illegible]

Date Sampled [illegible] Time Sampled [illegible]

RECEIVED DEC 2 1 1984

Line No.	Parameter	Units Of Measure	MDL	Value	Comments
	CONVENTIONALS				
1	Chloride	mg/l			
2	Fluoride	mg/l			
3	Nitrate as N	mg/l			
4	Sulfate as SO4	mg/l	2	22	
5	Phenolics, Total	mg/l			
6	Total Organic Halides (TOX)	ug/l			
	Total Organic Halides (TOX)	ug/l			
	Total Organic Halides (TOX)	ug/l			
	Total Organic Halides (TOX)	ug/l			
	[illegible]				
	[illegible]				
	[illegible]				
	[illegible]				
	[illegible]				
	[illegible] Lab	[illegible]			
	[illegible]				
	[illegible] Lab				
	pH Lab	std			
	[illegible] as CaCO3	mg/l			
	[illegible]				
	[illegible] as N	mg/l			
	[illegible] Total	[illegible]			
	[illegible]m, Fecal	[illegible]			
	[illegible] Apparent (Lab)				
	[illegible]de, Total				
	[illegible] Organic Carbon				
	[illegible]				
	[illegible]	TON			
33	Petroleum Hydrocarbons IR	mg/l			
	[illegible]				
35	Phosphate, Total	mg/l			
36	[illegible]	mg/l			
	[illegible]	mg/l			

Appendix E

Chain-of Custody Forms

1) A field Chain-of-Custody form (CC1) is included for all samples shipped by ETC shuttle.

2) An in-house sample Chain-of Custody form is included for the period the sample was in ETC's possession.

3) A subcontractor's Chain-of-Custody form is included for any analytical work not performed within ETC's laboratory.

4) Any additional Chain-of-Custody material provided by a client or by a client's sampling agent is also included.

CHAIN OF CUSTODY FORM (CC1)

Seal Date: 11/20/84
Sealed By: Herberg

SHIP TO:

Company: Marathon Petroleum Co
Attn.: Vicky May
Facility/Site: Marathon Ave
Address: Robinson, Ill 62454
Phone: ( ) -

SAMPLE IDENTIFICATION

Facility/Site Code: M|P|C|R|O|B|G|W|M|

Source Codes:

Well	(W)	River/Stream	(R)	Surface Impoundment	(I)	Lake/Ocean	(L)
Soil	(S)	Bottom Sediment	(B)	Pretreatment Facility	(P)	Treatment Facility	(T)
Outfall	(O)	Generation Point	(G)	Leachate Collection Sys.	(C)	Other	(X)

Source Code (from above)	Your Sample Point ID (left justify)	Start Date (mo/day/yr)	Start Time (2400 hr. clock)	Elapsed Hours (composite)
W	B4	112031284	1900	-1-
Example: W	10 1 J 2 6 3 P 1	05 15 81	0930	13

SHUTTLE CONTENTS

Sample Bottle	Condition	Sample Bottle	Condition
F9336 PH1-2		F9336 D1	All partially frozen
F9336 TX1		F9336 B1	
F9336 M1		F9336 C1	
F9336 O/G1		F9336 A1	

CHAIN OF CUSTODY CHRONICLE

1. Shuttle Opened By: (print) Vicki May Date: 11/28/84 Time: 3:30
   Signature: Vicki May Seal #: 0033831 Intact: +
2. I have received these materials in good condition from the above person.
   Name: Signature:
   Date: Time: Remarks:
3. I have received these materials in good condition from the above person.
   Name: Signature:
   Date: Time: Remarks:
4. I have received these materials in good condition from the above person.
   Name: Signature:
   Date: Time: Remarks:
5. Shuttle Sealed By: (print) VC May Date: 12/3/84 Time: 2PM
   Signature: VC May Seal #: 0033832

ETC USE ONLY Opened By: Date: 12-4-84 Time: 1830
Seal #: 33832 Condition: ok

ETC ENVIRONMENTAL TESTING and CERTIFICATION

F9336
ETC Sample No.:

SAMPLE POINT INFORMATION FORM (CC2)

FIELD MEASUREMENT DATA

Select up to 3 parameters to be recorded by entering the appropriate code letter in the first space for each of the 3 data entry fields provided. Enter the actual measurement data (in the units specified) for the three parameters you have selected.

PARAMETERS		ACTUAL (left justify)	EXAMPLE
Flow (CFS)	A		C 2 5
Volume (Gal)	B		
Sample Depth (Ft)	C		
Depth to Water (Ft)	D		F 5 0
Discharge Rate (GPM)	E		
Depth to Bottom (Ft)	F		
Event Time (2400-Hr Clock)	G		G 1 1 0 3
Depth to Screen (Ft)	H		

FIELD TEST DATA

DO (Mg/L)

Sample Temp. (F) 51.9

	Single Measurement	2nd of Quadruplicate	3rd of Quadruplicate	4th of Quadruplicate
pH	7.15	7.11	7.13	7.13
Specific Conductance (uMHOS/CM)				

THE FOLLOWING DATA IS FOR YOUR RECORDS ONLY

SAMPLING METHOD (choose one)

AIR-LIFT PUMP ( )	PERISTALTIC PUMP ( )	THIEF ( )
AUGER ( )	PETERSEN ( )	TRIER ( )
BAILER (X)	PISTON PUMP ( )	VEHMEYER ( )
BOTTLE ( )	SCOOP/SHOVEL ( )	OTHER _____
COLIWASA ( )	SQUEEZE PUMP ( )	_____
DIPPER ( )	SUBMERSIBLE PUMP ( )	_____
KEMMERER ( )	SUCTION LIFT PUMP ( )	
NISKIN ( )	SURBER ( )	

SAMPLE TYPE (choose one)

GRAB (X) COMPOSITE ( ) OTHER ( )

_____ _____

_____ _____

(describe) (describe)

WEATHER

_____

_____

_____

(describe)

SAMPLE DESCRIPTION (e.g. color, odor)

_____

_____

_____

(describe)

Form Prepared By: Vicki Mey (name (print)) Employer: Marathon

ETC Form CC2 6/1/82

ETC ENVIRONMENTAL TESTING and CERTIFICATION

7376

Name of Subcontractor Core

Please complete this document and return original with Sample Shuttle. Prepare copy for your records.

This shuttle contains:

ETC Sample numbers: F9336

To be analyzed for: Gross β

Data is required by 12/30/84. If deadline cannot be met, please notify John Hamilton (201) 225-5600.

85

Sample Shuttle sealed by: D. Schuckert

Date: 12/18/84 Time: 6:00 Seal Number: 31997

Sample Shuttle opened by: R. Dimond

Date: 12-19 Time: 4:40 Seal Number:

Was seal intact? Yes ✓ No

For return:

Sample Shuttle sealed by: R. Dimond

Date: 12-19 Time: 4:40 Seal Number: 31996

Sample Shuttle opened by: M Horberg

Date: 1/3/85 Time: 12 pm Seal Number: 0031996

Was seal intact? Yes ✓ No

7576

ETC ENVIRONMENTAL TESTING and CERTIFICATION

Request for Analysis

Name of Subcontractor: Chyun

ETC Sample Number(s) F9336

Send bill to: John Hamilton
Send report to: John Hamilton

ETC Corporation
284 Raritan Center Pkw.
Edison, NJ 08837
(201) 225-5600

Date Data Required: 12/14/84
If deadline cannot be met, contact John Hamilton immediately.

Please perform the analyses requested below:

- ___ Color
- ___ Conductance, Specific
- ___ Odor
- ___ pH
- ___ Turbidity
- ___ Total Solids
- ___ Total Suspended Solids
- ___ Total Dissolved Solids
- ___ Total Volatile Solids
- ___ Gross Alpha and Gross Beta*
- ___ Radium 226 if Gross Alpha exceeds 5 pCi/l
- ___ Radium 228 if Radium 226 exceeds 3 pCi/l
- ___ Coliform, Total
- ___ Coliform, Fecal
- ___ Biological Oxygen Demand (5 day, 20 degree C)
- ___ Chemical Oxygen Demand (COD)
- ___ Oil & Grease (Gravimetric)
- ___ Petroleum Hydrocarbons (Infrared)
- ___ Organic Carbon, Total (TOC)
- ___ Phenols, Total (as Phenolics)
- ___ Methylene Blue Active Substances (MBAS) (Foaming Agents, Surfactants)

* If Gross Alpha exceeds 5 pCi/l, John Hamitlon must be notified immediately.

- ___ Acidity
- ___ Alkalinity
- ___ Bromide
- ___ Chloride
- ___ Chlorine, Total Residual
- ___ Cyanide, Total
- ___ Ammonia (as N)
- ___ Total Kjeldahl Nitrogen (TKN)
- ___ Nitrate
- ___ Nitrate-Nitrite
- ___ Nitrite
- ___ Oxygen, Dissolved
- ___ Phosphorous, Ortho Phosphate
- ___ Silica, Dissolved
- X Sulfate (as SO_4)
- ___ Sulfide (as S)
- ___ Sulfite (as SO_3)
- ___ Fluoride

OTHERS

Sample(s) Relinquished by: Mark Kelly

Date 12/5/84 Time 4:30

Sample(s) Received by: Mark Kelly

Date 12/5/84 Time 4:30

Sample Number	Log Bank	Sample Vol. (ml)	Extract Vol. (ml) HERB		Comments	Analyst
F9759	7335	480	2.5			
F9760		465				
F9761		500				
F9762		485				
F8593	7304	490				
F8594		490				
F8595		500				
G1569	7340	425				
G0512	7363	500				
G1557	7362	475				
F9336	7376	470				
F9985	7373	500				
G2422	7378	475				
G2432		500				
G2485		485				
G2424	7399	500				
QC 2498		500	2.5			
QC 2498 S		500	2.5			
G1569 S		500	2.5			
G1557 R		500	2.5			

EXTRACTION LOG

QC Batch # 2498

Analysis DW/HERB/GC

Matrix H2O

Turnaround NORM

Date Start 12/20/84

Extraction Method:

Sep. Funnel ✓

Continuous

Other

COMMENTS:

SAMPLES: G2424, G2432, G2422, F9985, F9336, F8595, F8594, F8593 Extra Fluid GF

Spike verified: S McAuliffe

FRACTION	SPIKE			SURROGATES		
	Amt (ml)	Conc.	Lot #	Amt (ml)	Conc.	Lot #
2,4,5-TP	0.5	10 mg/ml	9291			
2,4-D	5.0	10 mg/ml	9407			

SET-UP: [signature]

CONC: [signature] 12/26/84

UPD/SUPERVISOR: Vicky Wheaton ✓ 12/26/84

	Log Link	Sample Vol. (ml)	Extract Vol. (ml) PEST		Comments	Analyst
84	7335	500	5.0 PEST			
90		500	5.0			
91		500	5.0			
92	↓	500	5.0			
93	7304	500	5.0			
94		500	5.0			
95	↓	500	5.0			
89	7340	500	5.0			
912	7363	500	5.0			
87	7362	500	5.0			
86	7376	500	5.0			
85	7373	500	5.0			
22	7370	500	5.0			
32		500	5.0			
2486	↓	500	5.0			
124	7399	500	5.0			
2499		500	5.0			
2499	S	500	5.0			
2491	S	475	5.0			
2512	R	485	5.0			

Extraction [illegible]

QC Batch # 2499

Analysis DW/PST/GC

Matrix H2O

Turnaround NORM

Date Start 12/20/84

Extraction Method:

Sep. Funnel ✓

Continuous

Other

COMMENTS:

Spikes Verified:
BG 12/20/84

EXTRACTION	SPIKE			SURROGATES		
	Amt (ml)	Conc.	Lot #	Amt (ml)	Conc.	Lot #
ANE	2.0	1.0 UG/ML	9403			
APHENE	2.5	1.0 UG/ML	9287			
IN	0.1	1.0 UG/ML	9034			
LOR/CHLOR	5.0	10.0 UG/ML	9404			

Set-up: [signature] 12/20/84

UPD/SUPERVISOR: Udyawkiawon 12/21/84

CONC: Dave Gallante 12/21/84

TEFLON LID ON SAMPLE JAR

```
EC 27, 1984    11:40
EQUENCE: HRB8        ON CRN 15
HANNEL  8

JBSEQUENCE  1

METHOD
HERB8

DIALG-PRG  PARAM-FILE
'N

#WSHS  #PMPS  STOP
  5,     5,    1

ISO  POST-BTL#  POST-#WSHS
40 ,      0,          1

SAMPLES
     SAMPLE-NAME    BTL# PROC-FILE  RAW-FILE   %DIL-F  STD-AMT  SMP-AMT
 1   24D 24STP B ,    1, P9506 :PK, R9506 :PK, 100.00,  1.0000,  1.0000
 2   QC2498      ,    2, P9507 :PK, R9507 :PK, 100.00,  1.0000,  1.0000
 3   QC2498S     ,    3, P9508 :PK, R9508 :PK, 100.00,  1.0000,  1.0000
 4   G1569S      ,    4, P9509 :PK, R9509 :PK, 100.00,  1.0000,  1.0000
 5   G1557R      ,    5, P9510 :PK, R9510 :PK, 100.00,  1.0000,  1.0000
 6   F9759-      ,    6, F9759A:PK, F9759B:PK, 100.00,  1.0000,  1.0000
 7   F9760-      ,    7, F9760A:PK, F9760B:PK, 100.00,  1.0000,  1.0000
 8   F9761-      ,    8, F9761A:PK, F9761B:PK, 100.00,  1.0000,  1.0000
 9   F9762-      ,    9, F9762A:PK, F9762B:PK, 100.00,  1.0000,  1.0000
10   F8593-      ,   10, F8593A:PK, F8593B:PK, 100.00,  1.0000,  1.0000
11   F8594-      ,   11, F8594A:PK, F8594B:PK, 100.00,  1.0000,  1.0000
12   24D 24STP A ,   12, P9511 :PK, R9511 :PK, 100.00,  1.0000,  1.0000
13   F8595-      ,   13, F8595A:PK, F8595B:PK, 100.00,  1.0000,  1.0000
14   G1569-      ,   14, G1569A:PK, G1569B:PK, 100.00,  1.0000,  1.0000
15   G0512-      ,   15, G0512A:PK, G0512B:PK, 100.00,  1.0000,  1.0000
16   G1557-      ,   16, G1557A:PK, G1557B:PK, 100.00,  1.0000,  1.0000
17   F9336-      ,   17, F9336A:PK, F9336B:PK, 100.00,  1.0000,  1.0000
18   F9985-      ,   18, F9985A:PK, F9985B:PK, 100.00,  1.0000,  1.0000
19   G2422-      ,   19, G2422A:PK, G2422B:PK, 100.00,  1.0000,  1.0000
20   G2432-      ,   20, G2432A:PK, G2432B:PK, 100.00,  1.0000,  1.0000
21   G2435-      ,   21, G2435A:PK, G2435B:PK, 100.00,  1.0000,  1.0000
22   G2424-      ,   22, G2424A:PK, G2424B:PK, 100.00,  1.0000,  1.0000
23   24D 24STPC  ,   23, P9512 :PK, R9512 :PK, 100.00,  1.0000,  1.0000
24   /E
```

```
HOD
ST8

LG-PRG  PARAM-FILE


HS  #PMPS  STOP
      5,     1

  POST-BTL#  POST-#WSHS
       0,          1

PLES
SAMPLE-NAME     BTL# PROC-FILE   RAW-FILE    XDIL-F  STD-AMT  SMP-AMT
DW STDB      ,    1, P9513 :PK, R9513 :PK, 100.00,  1.0000,  1.0000
QC2499       ,    2, P9514 :PK, R9514 :PK, 100.00,  1.0000,  1.0000
QC2499S      ,    3, P9515 :PK, R9515 :PK, 100.00,  1.0000,  1.0000
F9761S       ,    4, P9516 :PK, R9516 :PK, 100.00,  1.0000,  1.0000
G0512R       ,    5, P9517 :PK, R9517 :PK, 100.00,  1.0000,  1.0000
F9759        ,    6, F9759C:PK, F9759D:PK, 100.00,  1.0000,  1.0000
F9760        ,    7, F9760C:PK, F9760D:PK, 100.00,  1.0000,  1.0000
F9761        ,    8, F9761C:PK, F9761D:PK, 100.00,  1.0000,  1.0000
F9762        ,    9, F9762C:PK, F9762D:PK, 100.00,  1.0000,  1.0000
F8593        ,   10, F8593C:PK, F8593D:PK, 100.00,  1.0000,  1.0000
F8594        ,   11, F8594C:PK, F8594D:PK, 100.00,  1.0000,  1.0000
DW STD A     ,   12, P9518 :PK, R9518 :PK, 100.00,  1.0000,  1.0000
F8595        ,   13, F8595C:PK, F8595D:PK, 100.00,  1.0000,  1.0000
G1569        ,   14, G1569C:PK, G1569D:PK, 100.00,  1.0000,  1.0000
G0512        ,   15, G0512C:PK, G0512D:PK, 100.00,  1.0000,  1.0000
G1557        ,   16, G1557C:PK, G1557D:PK, 100.00,  1.0000,  1.0000
F9336        ,   17, F9336C:PK, F9336D:PK, 100.00,  1.0000,  1.0000
F9985        ,   18, F9985C:PK, F9985D:PK, 100.00,  1.0000,  1.0000
G2422        ,   19, G2422C:PK, G2422D:PK, 100.00,  1.0000,  1.0000
G2432        ,   20, G2432C:PK, G2432D:PK, 100.00,  1.0000,  1.0000
G2435        ,   21, G2435C:PK, G2435D:PK, 100.00,  1.0000,  1.0000
G2424        ,   22, G2424C:PK, G2424D:PK, 100.00,  1.0000,  1.0000
DW STDC      ,   23, P9519 :PK, R9519 :PK, 100.00,  1.0000,  1.0000
/E
```

ETC ENVIRONMENTAL TESTING and CERTIFICATION

LL 7376

Metals Analysis Custody Log

Samples F 9336

	Chemist	Date
Hg Prep	Joan Komarek	1/8/85
AA/ICAP Prep	Maura Jan McClure	1/7/85

Lab Supervisor _____ date _____

ANALYSIS/CUSTODY

TITLE: [illegible]

BOOK NO. CV4 PAGE NO. [illegible]

	SAMPLE #	CONC.
1	E[illegible]9	1590
2	E[illegible]5	3090
3	E[illegible]1	3140
4	E[illegible]2	2360
5	E[illegible]5	2220 1:1000
6	G2435	re-run straight 1:10
7	G2433	14.8 1:10
8	G2554	3.87 1:10
9	G2429	KH SEE SPIKE DATA
10	G3614	5.96 1:10
11	G2544	5.66 1:10
12	G2545	6.23 1:10
13	G1843	18.3 1:10
14	G1844	50.5
15	G1845	66.3 1:1000
16	F8970	1680
17	F8971	102 KH 1:10
18	F8972	1420 1:1000
19	F8973	167
20	F8974	1240 1:1000
21	G0687	165.8
22	G0690	26.9
23	G0513	38.7
24	G0514	22.1
25	G0515	37.3
26	G0516	14.5 1:10
27	G0517	31.8
28	G0518	13.9 1:10
29	G0519	24.5

	SAMPLE #	CONC
30	G0520	KH SEE SPIKE DATA
31	G0521	re-run 1:10
32	G0522	31.3
33	G0523	25.0
34	G2568	22.7 1:10
35	G2567	16.1
36	G1791	88.7
38	G1792	129
38	G1793	103
39	G1794	136
40	G1795	147
41	F9860	re-run straight 1:1,000
42	G3425	3040
43	G3683	re-run 1:10
44	G3684	30.8 1:10
45	G3685	2.72 1:10
46	G3686	4.95
47	G3687	re-run straight
48	G1557	re-run straight
49	KH [illegible]	KH
50	F9985	SEE SPIKE DATA
51	G3688	29.3
52	[illegible]	re-run KH straight
53	G2429	8.80 1:10
54	F9336	20.3 1:10
55		
56		
57		
58		

UNITS: MG/L Detection limit = 0.2 KH mg/l

COMMENTS:

< = less than 0.2 mg/l

All samples run at 1:10 dilution unless noted otherwise. KH

Blank Recovery = 4.21/4.00 = 105%.

EPA Std. Rec. = ① 9.65/8.90 = 108%.
② 9.44/8.90 = 106%.

SPIKE RECOVERY

SAMPLE #:	G2429	G0520	KH G F9985
REPLICATE 1:	0.303	1.35	0.165
REPLICATE 2:	0.270	1.26	0.205
MEAN :	0.287	1.31	0.185
STD. DEV. :			
SPIKE VALUE:	4.00	4.00 KH 5.58	4.00
REPLICATE 1:	4.50	5.58	4.31
REPLICATE 2:			
MEAN :			
STD. DEV. :			
% RECOVERY :	105%	107%	103%

REPLICATE DATA:

SAMPLE # :			
REPLICATE 1:			
REPLICATE 2:			
MEAN :			
STD DEV. :			

ANALYST: Karen Hudak

DATE : 1/7/85

VERIFIED BY: [signature] 1/8/85

ANALYSIS/CUSTODY

(29 of 87)

TITLE: Fluoride BOOK NO. CV4 PAGE NO. 41

	SAMPLE #	CONC.
1	G1558	BMDL
2	G1559	BMDL *
3	G1560	0.396
4	G1561	BMDL
5	F9759	0.128
6	F9760	0.139
7	F9761	0.174
8	F9762	BMDL *
9	F9959	0.177
10	F9960	0.159 (ave) *
11	F9963	0.126
12	G0526	BMDL
13	G0535	0.204
14	G1562	0.307
15	G1563	0.468
16	G1564	0.408
17	G1565	0.355
18	G1566	0.291
19	G1567	0.473
20	G1568	0.416
21	G1569	0.380
22	G0512	0.164
23	G1557	BMDL
24	F9336	0.341
25	F9985	0.633
26	G2422	0.148
27	G2432	BMDL
28	G2435	BMDL
29	G2861	0.108

	SAMPLE #	CONC
30	G2862	BMDL
31	G2863	0.121
32	G2864	BMDL
33	G2865	BMDL
34	G2866	BMDL
35	G2424	0.131
36	G2425	0.139
37	G2428	BMDL
38	G2430	BMDL
39	G2434	BMDL
40	F8593	BMDL **
41	F8594	0.510 **
42	F8595	0.585 **
43		
44		
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57		
58		

UNITS: MG/L Detection limit = 0.1 mg/l

COMMENTS:

BMDL = less than 0.1 mg/l

* G1559, F9762, ~~F9960~~ for Run at 1:2 dilution due to matrix interferences on previous run (MDL = 0.2)

** F8593, 94, 95 Run at 1:5 dilution due to matrix interferences when run straight and at 1:2 (MDL = 0.5)

Blank Recovery = 1.06/1.00 = 106%

EPA Std. Rec. = 0.383/0.38 = 101%

SPIKE RECOVERY

SAMPLE #:	F9960	G1557	G2435
REPLICATE 1:	0.156	BMDL	ND
REPLICATE 2:	0.162	BMDL	BMDL
MEAN :	0.159	BMDL	BMDL
STD. DEV. :			
SPIKE VALUE:	1.00	1.00	1.00
REPLICATE 1:	1.15	1.05	0.994
REPLICATE 2:			
MEAN :			
STD. DEV. :			
% RECOVERY :	102%	105%	99%

REPLICATE DATA:

SAMPLE # :			
REPLICATE 1:			
REPLICATE 2:			
MEAN :			
STD DEV. :			

ANALYST: SMcAdam

DATE : 12/16/84

VERIFIED BY: 12/17/84

ANALYSIS/CUSTODY

TITLE: NITRATE BOOK NO. CV4 PAGE NO. 50

	SAMPLE #	CONC.
1	G-1564	0.498
2	G-1565	0.562
3	G-1568	0.546
4	G-1756	ND
5	G-1757	0.760
6	G-2435	0.134
7	F9336	ND
8	F9985	1.38
9	G-2422	BMDL
10	G2432	BMDL
11	G-1557	BMDL
12	G-0512	BMDL
13	FF138	BMDL
14	FF141	0.115
15	F9942	BMDL
16	FF144	0.395
17	FF145	0.851
18	FF146	0.946
19	G2424	BMDL
20	G2472	ND
21	G2428	0.145
22	G2430	BMDL
23	G-2434	0.248
24	G2470	0.769
25	G2471	ND
26	G-2425	0.163
27	G2573	ND
28	G2433	0.127
29	F8595	BMDL

	SAMPLE #	CONC
30	G1563	0.521 *
31	F9959	0.531 *
32	F9960	1.44 *
33	G0535	0.866 *
34		
35		
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57		
58		

UNITS: MG/L Detection limit = 0.1 mg/l

COMMENTS:

< = less than 0.1 mg/l BMDL

* = 1:10 DILUTION

DATA FROM RUN #2

Blank Recovery = 0.435/0.4 = 109 %

EPA Std. Rec. = 0.0976/0.08 = 122 %

SPIKE RECOVERY

SAMPLE #:	G-1557	G2425	
REPLICATE 1:	BMDL	0.178	
REPLICATE 2:	BMDL	0.148	
MEAN :	BMDL	0.163	
STD. DEV. :			
SPIKE VALUE:	0.4	0.4	
REPLICATE 1:	0.410	0.325	
REPLICATE 2:			
MEAN :			
STD. DEV. :			
% RECOVERY :	103	81.3	

REPLICATE DATA:

SAMPLE # :			
REPLICATE 1:			
REPLICATE 2:			
MEAN :			
STD DEV. :			

ANALYST: [signature]

DATE : 12/29/04

VERIFIED BY: [signature] 1/2/05

ANALYSIS/CUSTODY

TITLE: PHENOL BOOK NO. CV4 PAGE NO. 52

	SAMPLE #	CONC.
1	G0596 BG	BMDL
2	G0798	(1:100) 0.107
3	G0799	(1:50) 0.128
4	G1563	*
5	G1564	*
6	G1565	0.124
7	G1566	0.121
8	G1567	0.153
9	G1568	0.105
10	G1569	*
11	F7892	(1:100) .0942
12	F7898	RE-RUN @ 1:50
13	F7899	RE-RUN @ 1:50
14	F7900	RE-RUN @ 1:100
15	F7901	(1:100) 0.0942
16	G1557	BMDL
17	F9336	BMDL
18	F9985	BMDL
19	G2422	BMDL
20	G2432	BMDL
21	G2435	(RE-RUN 0.143 SPIKE)
22	F9761	*
23	G3065	(RE-RUN .060 DIST.)
24	G2424	RE-RUN STRAT. 0.120
25	G2425	RE-RUN STRAT. 0.176
26	G2428	BMDL
27	G2430	BMDL
28	G2437	BMDL
29	G0665	BMDL

	SAMPLE #	CONC
30	G2573	0.0713
31	G2433	0.0644
32	G2429	0.0636
33	G3064	0.0534
34		
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UNITS: MG/L Detection limit = 0.005 mg/l

COMMENTS:

BMDL = less than 005 mg/l

* RE-RUN STRAIGHT; AIR SPIKE PROBABLE CAUSE OF OFF SCALE VALUE

** REPLICATE NOT AVAILABLE BECAUSE OF AIR SPIKES OCCURING WHEN SAMPLE WAS RUN.

G0798, 99; Reanalyze to verify high levels JH 1/6/95

Blank Recovery = 0.200/0.2 = 100%

EPA Std. Rec. = 0.149/0.136 = 110%

SPIKE RECOVERY

SAMPLE #:	G2428	G1557	
REPLICATE 1:	BMDL	BMDL	
REPLICATE 2:	BMDL	**	
MEAN :	BMDL		
STD. DEV. :			
SPIKE VALUE:	0.2	0.2	
REPLICATE 1:	0.208	0.221	
REPLICATE 2:			
MEAN :			
STD. DEV. :			
% RECOVERY :	104%	111	

REPLICATE DATA:

SAMPLE # :			
REPLICATE 1:			
REPLICATE 2:			
MEAN :			
STD DEV. :			

ANALYST: [signature]

DATE : 12/30/94

VERIFIED BY: [signature] 1/3/95

Project No._____

Book No._____ TITLE TOC

From Page No.___

Log #	Sample #	TOC
13-76	F9336	16.58
		16.58
		16.18
		16.49
	F9337	35.29
		36.09
		35.88
		35.65
	400 ppm	403.7
73	F9985	6.590
		5.413
		6.015
		6.138
414	F9220	2.226
		2.735
	F9222	6.407
		6.851
	F9223	2.064
		1.640
	F9224	1.962
		1.784
	400 ppm	408.3
	F9225	2.348
		1.325
	F9226	1.427
		1.245
	F9227	1.696
		1.570
	F9228	2.228
		1.691
	F9229	2.354
		1.619
	F9230	3.340
		2.867
	F9231	2.580
		3.364

Log #	Sample #	TOC
	400 ppm	407.4
7340	G1562	1.458
		1.356
		1.730
		1.528
	G1563	2.500
		2.408
		2.707
		2.647
	G1564	1.446
		1.583
		1.472
		1.887
	400 ppm	413.8
Low Spike	G1564-S	21.28
		21.77
High Spke	F9337-S	202.0
		202.3

To Page No.____

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by S. McArthur	12/29/84

From Page No. ____

#1

meth. blk -0.50
1.39
Cal blk .90
cal std. 5.89

99.8 %

G1770

T 5.76 > 35.2
B 2.54
T 4.61 > 41.1
B 2.28
T 3.46 > 41.0
B 3.42
T 2.83 > 14.4
B 0.06
flushed cell
meth blk 1.40
Cal blk 0.63
cal std. 5.99

107 %

G1772

T 4.30 > 29.0
B 1.14
T 4.57 > 31.7
B 1.29
T 5.19 > 39.3
B 1.52
T 4.42 > 30.2
B 1.07
flushed cell

$\bar{X}$ mb = 1.39
$\bar{X}$ Cb = 0.765

#2

meth. blk 0.36
cal. blk 0.85
cal std 4.25
4.68
4.98

82.6 %

G1771

T 2.83 > 30.3
B 1.69
T 4.01 > 55
B 2.98
T 3.32 > 29
B 1.07
T 3.75 > 37.3
B 1.47
flushed cell
meth blk 1.13
cal blk 0.59
cal std. 4.81

84.4 %

G1557

T 1.36 > 2.3
B 0.54
T 1.50 > 3.7
B 0.63
T 1.62 > 4.9
B 0.59
T 1.89 > 7.6
B 0.71
flushed cell

$\bar{X}$ mb = 0.145
$\bar{X}$ Cb = 0.72

#3

meth blk 0.71
1.60
Cal blk -0.01
1.71
cal std. 6.65

G1769

91 mls
T 3.77 > 33.8
B 1.41
T 4.26 > 39.4
B 1.89
T 3.31 > 25.1
B 2.46
T 5.64 > 48.
B 2.41
flushed cell
meth blk. 1.61
Cal blk 0.61
cal std. 6.33

114.4 %

F9336

T 2.29 > 6.8
B 0.99
T 2.68 > 10.7
B 1.34
T 2.67 > 10.6
B 0.89
T 2.48 > 8.7
B 0.99
flushed cell

$\bar{X}$ mb = 1.605
$\bar{X}$ Cb = 1.16

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To Page No. ____

Witnessed & Understood by me	Date	Invented by D. Ballarte (A.M.)	Date
		Recorded by	

TITLE PH / SCOND

Project No.

Book No.

From Page No.

Sample	PH	Temp.	KM	K	Range	Scond
G5536	8.0	15.8	0.372	0.451	2mV	450
"	8.0	15.8	0.370	0.448	"	450
F9336	7.8	13.3	0.398	0.512	"	500
"	7.8	13.2	0.401	0.518	"	500
"	7.8	13.2	0.407	0.525	"	500
"	7.8	13.2	0.409	0.528	"	500
F8567	7.6	14.9	2.03	2.515	20mV	2500
"	7.6	14.9	2.08	2.58	"	2600
G4698	7.6	16.5	0.155	0.185	2mV	190
"	7.6	16.5	0.153	0.182	"	180
G5535	7.2	12.1	4.15	5.51	20mV	5600
"	7.2	12.2	4.24	5.61	"	5600
G4223	7.4	14.8	0.707	0.878	2mV	880
"	7.4	14.8	0.710	0.882	"	880
"	7.4	14.8	0.724	0.899	"	890
"	7.4	14.8	0.708	0.879	"	880
G4481	7.0	16.5	41.8	49.9	200μV	7100
"	7.0	16.5	41.9	50.0	"	7100
F9538	7.3	18.5	30.3	34.6	200mV	34000
"	7.2	18.3	29.5	33.8	"	34000
G4235	7.9	21.7	0.505	0.538	2mV	540
"	7.8	21.6	0.498	0.531	"	540
F8659	8.2	17.1	0.345	0.406	"	400
"	8.2	17.1	0.348	0.410	"	
"	8.2	17.1	0.356	0.419	"	
"	8.2	17.1	0.366	0.431	"	
G5026	7.6	19.8	0.582	0.686	2mV	<100
"	7.6	19.8	0.580	0.683	"	<100

Mix 1:1 w/ H2O

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FEB 22 1985

IEPA-DLPC

End of Text

To Page No.

Witnessed & Understood by me, 1/29/85

Date

Invented by

Date

Recorded by Greg Mercer 1/29/85

Technical Report

for

MARATHON PETROLEUM COMPANY

MARATHON AVE.

ROBINSON, IL 62454

Chain of Custody Data Required for ETC Data Management Summary Reports

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
G4527	MARATHON PETROLEUM COMPANY	MPCROBGWM	FIELD N-2	841219		

Denis C. K. Lin, Ph.D.
Vice President
Research and Operations

TABLE OF CONTENTS

Introduction

This report contains the analytical results on your water sample, FIELD N-2 84/12/19. It is designed to include comprehensive data from the entire analytical process in order to satisfy the needs of various levels of review.

The results obtained from your sample are presented in tabular format immediately following this introduction. Quality assurance data is tabulated along with the appropriate sample results for verification. Depending on the analyses ordered, the quality assurance data may include results from blank, spiked blank, spiked sample (i.e. matrix spike) and replicate sample as well as results from surrogate compound analyses. Quality assurance data for verification of proper instrument performance is also included where appropriate. The report appendices include the chain of custody record for your sample and, where appropriate, the gas chromatograms and mass spectra.

The procedures used in the analysis of the sample are described in this report's methodology section. All analytical procedures within our laboratory are performed within a strictly enforced Quality Assurance Protocol. A description of this Protocol is included in the report.

Results

Sample results, and associated quality assurance data, are always tabulated in one or more of this report's Quantitative Results Tables. The format of each table varies with the class of analysis.

ETC ENVIRONMENTAL TESTING and CERTIFICATION

FEB 6, 1985

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA

Ground Water Monitoring - Conventional Analysis Data (QR10)

Chain of Custody Data Required for ETC Data Management Summary Reports

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
G4527	MARATHON PETROLEUM COMPANY	MPCROBGWM	FIELD N-2	841219		

NPDES Number	Compound	Results: Sample Concen. ug/l	Results: MDL ug/l
	Total Organic Halides (TOX)	15	5
	Total Organic Halides (TOX)	28	5
	Total Organic Halides (TOX)	36	5
	Total Organic Halides (TOX)	48	5

Methodology
for
Analysis of Total Organic Halides

The method employed in the analysis of your sample for Total Organic Halide (TOX) is the established EPA Method 450.1, November, 1980. This is identical to EPA Method 8.66 in SW-846, 2nd Ed., 7/82 for evaluation of TOX in drinking and ground waters as provided under 40 CFR 265.92.

The EPA TOX method, in summary, can be described as follows: A sample of water that has been protected against the loss of volatiles by the elimination of headspace in the sampling container, and is free of undissolved solids, is passed through a column containing 40 mg of activated carbon. The column is washed to remove any trapped inorganic halides, and is then pyrolized to convert the absorbed organohalides to a titratable species which is then measured by a microcoulometric detector.

Summary of
Quality Assurance/Quality Control Procedures (QA/QC)

ETC bases its quality assurance protocols on the following government guidelines:

- "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA-600/4-79-019, March 1979;

- National Enforcement Investigation Center Policies, and Procedures manual; EPA-330/9/79/001-R, October 1979;

- the recommended guidelines for EPA Methods 624 and 625. (Federal Register, December 3, 1979, pp. 69532-69559);

- "Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples," EPA 600/8-80-038, June 1980; and

- "Determination of 2,3,7,8-TCDD in Soil and Sediment" EPA, Region VII, Kansas City, September 1983.

- Organic Analysis: Multi-media, Multi Concentration-IFB WA84-A267

- Dioxin Analysis:Soil/Sediment Matrix; Multi-Concentration; Selected Ion Monitoring with Jar Extration Procedure-IFB WA84-A002

However, we have modified our protocols to provide a higher level of QA/QC than the guidelines require. For example, we analyze a higher than required number of quality control samples and we pay especially careful attention to the certification of the "reference standard" compounds we use in analysis. Below are listed the key QA/QC elements for the methods we used.

Analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry

- Each batch of 13 samples consists of 9 customer samples (at a maximun). one blank sample, one spiked blank, one spiked sample and one replicate sample. This amounts to a 30% quality control factor.

- Three surrogate compounds are added to each sample in the batch of 13.

- A blind quality control sample is introduced to the laboratory for analysis on a weekly basis.

- Each GC/MS is checked and retuned, if necessary, at the beginning of each day to ensure that its performance on bromofluorobenzene (BFB) meets the EPA criteria.

- A calibration curve for quantitation is prepared using a mixture of Volatile Organic Priority Pollutant "standards" at a minimum of 3 different concentrations and using a mixture of 3 internal standards at a constant concentration.

- The calibration curve is verified with a mixture of priority pollutant standards every day. If the response factors factors vary greater than 25%, the instrument must be recalibrated.

Analysis of Organic Compounds Extracted in Acid or Base/Neutral Solutions by Gas Chromatography/Mass Spectrometry

- Each batch of 20 samples consists of 16 customer samples (at a maximum), one blank sample, one spiked blank (for water matrices), one sample spiked with the priority pollutant standard mixture and a duplicate customer sample. This amounts to a 20% quality control factor.

- Three surrogate compounds are added to each sample in the batch for Base/Neutral analysis.
- Three surrogate compounds are added to each sample in the batch for Acid analysis.
- A blind quality control sample is introduced to the laboratory for analysis on a weekly basis.
- Each GC/MS is checked and retuned, if necessary, at the beginning of each day to ensure that its performance on decafluorotriphenylphosphine (DFTPP) meets the EPA criteria.
- A calibration curve for quantitation is prepared using a mixture of standards composed of either the Organic Acid or Base/Neutral Extractable Compounds at a minimum of 3 concentrations and using five internal standards for quantitation.

Analysis of Metals

All Samples

- New standards are prepared for each batch of samples.
- Normal calibration is performed using a blank sample and four standards that have been through the sample preparation procedure. A regression analysis is used to construct the calibration curve.
- All EP Toxicity samples and all samples determined by furnace atomic absorption are calculated by the "method of additions".
- For each sample analysis that requires the use of the "method of additions" technique, a three point calibration is performed using U.S. EPA "Methods for Chemical Analysis of Water and Wastes, 1979". Results are obtained using linear regression analysis. Any regression with a coefficient of correlation below 0.990 is considered suspect, necessitating review of calibration data or sample re-analysis.
- In constructing the normal calibration curves the lowest concentration levels we use are values greater than or equal to 5 times the Instrumental Detection Limit (IDL).
- All calibration standards are analyzed in duplicate, at a minimum.
- Independent reference standards are used to check the accuracy of calibration standards.
- A check standard is analyzed every ten samples to validate the normal calibration curve.
- One customer sample out of every ten is analyzed in triplicate.

Homogeneous Samples (except for Mercury analysis)

Samples are analyzed in batches of 30 or less. For batches in which the sample matrices are homogeneous, the QC program is a minimum of 25% and consists of analyzing:

- 3 sets of triplicate analyses;
- 2 Replicate spikes;
- 1 independent reference standard;
- 4 Calibration standards (processed using the sample preparation method);
- 4 Calibration standards (without sample preparation); and
- 1 Reagent Blank.

Heterogeneous Samples (except for Mercury analysis)

Samples are analyzed in batches of 30 or less. For batches in which the sample matrices are heterogeneous, the QC program is a minimum of 35% and consists of analyzing:

- 3 sets of triplicate analyses;
- 2 Replicate spikes;
- 1 Replicate independent reference standards;
- 4 Calibration standards (processed using the sample preparation method);
- 1 Procedural Blank;
- 4 Calibration standards (without sample preparation); and
- 1 Reagent Blank.

Analysis of Mercury

To analyze samples for mercury we group them by matrix in batches of 20 or less. Our QC program is a minimum of 30% and consists of analyzing:

- each of the 30 customer samples in duplicate;
- 3 sets of triplicate anaylses;
- 2 Replicate spikes;
- 2 Replicate independent reference standards;
- 10 Calibration standards (processed using the sample preparation method); and
- 2 Procedural Blanks.

Analysis of Pesticides, Herbicides and PCB's by Gas Chromatography

Pesticide, herbicide and PCB samples are grouped in batches of 16 customer samples or less according to the type of analysis to be performed. The QC program for each of these three types of analyses is a minimum of 20% and consists of analyzing:

- 1 procedural blank sample(a reagent blank is analyzed in the case of non-water matrices);
- 1 spiked blank sample (the spiked blank is eliminated in the case of non-water matrices);
- 1 replicate sample;
- 1 replicate spiked sample; and
- 1 known refernece QC sample for at least each 100 samples analyzed.

The instrument is calibrated each run with three standards, and checked every 10 samples.

Analysis of Cyanides, Phenols, Fluoride, Chloride, Nitrate and Nitrite

- All parameters are analyzed using a Technicon Autoanalyzer II GT.
- 3 calibration standards are analyzed at the beginning and end of each batch.

- Each batch (up to 80 samples) consists of analyzing one blank, one spiked blank, one duplicate and spiked sample every 20 samples, and an EPA known reference sample.

Analysis of Total Organic Carbon (TOC)

TOC samples are analyzed on a daily basis with the number of samples analyzed per day dependent on the request for duplicate or quadruplicate analyses. The quality control program is designed to maintain the appropriate amount of QC and consists of the following elements:

- Daily instrument calibration
- One blank
- Standard recalibration every 10 samples
- Spiked samples at a low and high level
- Every sample is run in duplicate at a minimum

Analysis of Total Organic Halide (TOX)

- Blank reagent water for absolute carbon backround must contain less than 5 ug/l of halide (as chloride).
- Using a trichlorophenol standard, the mean adsorption efficiency must be within +/- 15% of the standard value.
- Calibration standards are run every 10 samples.
- Every sample is run in duplicate at a minimum.

Analysis of 2,3,7,8-TCDD (Dioxin) by GC/MS (SIM)

- Each sample is dosed with a known quantity of $^{13}C_{12}$-2,3,7,8-TCDD as internal standard and $^{37}Cl_4$-TCDD as surogate standard. The action limits for surrogate standard results is +/- 40% of the true value. Samples showing surrogate standard results outside of these limits are reextracted and reanalyzed.
- Two laboratory "method blanks" are run along with each set of 24 or fewer samples. The method blank is also dosed with the internal standard and surrrogate standard
- At least one per set of 24 samples is run in duplicate to determine intralaboratory precision.
- Qualitative Requirements. The following are met in order to confirm the presence of native 2,3,7,8-TCDD:

  a. Isomer specificity must be demonstrated initially and verified once per 8-hour work shift. The verification consists of injecting a mixture containing TCDD isomers which elute close to 2,3,7,8-TCDD. The 2,3,7,8-TCDD must be separated from interferring isomers, with no more than 25% valley relative to the 2,3,7,8-TCDD peak.

  b. The 320/322 ratio is within the range of 0.67 to 0.87.

  c. Ions 320, 322, and 257 are all present and maximize together the signal to mean noise ratio must be 2.5 to 1 or better for all 3 ions.

  d. The retention time is equal (within 3 seconds) the retention time for the isotopically labeled 2,3,7,8-TCDD.

  e. At least one of the positives can be confirmed by obtaining partial scan spectra from mass 150 to mass 350. The partial scan guidelines are as follows:

. the 320/324 ratio should be 1.58 +/- 0.16

. the 257/259 ratio should be 1.03 +/- 0.10

. the 194/196 ratio should be 1.54 +/- 0.15

- One sample is spiked with native 2,3,7,8-TCDD at a level of 1.0 PPB (for soil) for each set of 24 or fewer samples.
- In cases where no native 2,3,7,8-TCDD is detected, the actual detection limit is estimated and reported based on a signal to noise ratio of 2.5 to 1 at ions 320 and 322.
- For each sample, the internal standard is present with at least a 10 to 1 signal to noise ratio for both mass 332 and mass 334. Also, the internal standard 332/334 ratio must be within the range of 0.67 to 0.87.

Subcontractor QA/QC

Each subcontractoer is required to maintain an appropriate level of quality control. To insure this, each subcontractor is required to submit to ETC the quality control data for all analyses it performs. This data is kept on file at ETC. In general, the amount of quality control required is one duplicate sample with one spiked sample for every ten analyses.

Chain-of-Custody

The chain-of-custody procedure is part of our quality assurance protocol. We believe our chain-of-custody record fully complies with the legal requirements of federal, state and local government agencies and of the courts of law. The record covers:

- labeling of sample bottles, packing the Sample Shuttle and transferring the Shuttle under seal to the custody of a shipper;
- outgoing shipping manifests;
- the chain-of-custody form completed by the person(s) breaking the Shuttle seal, taking the sample, resealing the Shuttle and transferring custody to a shipper;
- incoming shipping manifests;
- breaking the Shuttle's reseal;
- storing each labeled sample bottle in a secured area;
- disposition of each sample to an analyst or technician; and
- the use of the sample in each bottle in a testing procedure appropriate to the intended purpose of the sample.

The records show for each link in this process:

- the person with custody; and
- the time and date each person accepted or relinquished custody.

Appendix E

Chain-of Custody Forms

1) A field Chain-of-Custody form (CC1) is included for all samples shipped by ETC shuttle.

2) An in-house sample Chain-of Custody form is included for the period the sample was in ETC's possession.

3) A subcontractor's Chain-of-Custody form is included for any analytical work not performed within ETC's laboratory.

4) Any additional Chain-of-Custody material provided by a client or by a client's sampling agent is also included.

ETC ENVIRONMENTAL TESTING and CERTIFICATION

CHAIN OF CUSTODY

Company: Marathon Petroleum Co. Job No. _____

Address _____

_____

Attention: _____

Sample Description: rec'd 1 sealed cooler.

Customer ID	Description	ETC #
Field # N-2 Groundwater Marathon Petroleum 12/19/84 TOX in Quad.	(1) 1l	G4527

Sample(s) Relinquished by: Burlington Air Freight

Time: 10:00 Date: 12/20/84

Sample(s) Received by: BComito

Time: 10:00 Date: 12/20/84

MARATHON OIL COMPANY
ILLINOIS REFINING DIVISION
ROBINSON, ILLINOIS 62454
SAMPLING, CHAIN OF CUSTODY AND ANALYSIS RECORD
FOR RCRA GROUND WATER MONITORING PROGRAMS

Personnel Responsible for Sampling: Vicki May

Field Measurements

Well Identification N-2
Date of Sampling 12/19/84
Time 8:00 AM
Depth to Water 38"
Datum and Elevation —
Ground Water Elevation —
Weather Conditions Cloudy + drizzly, cool

Temperature 60°F
Sampling Equipment Pump - Bailer
Casing Volumes Removed 4
pH 6.9
Conductance (µmhos) 364
Comments

Sample Preservation and Analyses

Check Samples Shipped	Ref. No.	Container	Preservative	Parameters for Analysis
____	1	500 ml plastic	2.5 ml HNO_3 (Total Recoverable)	As, Ba, Cd, Cr, Pb, Hg, Ag, Se, Fe, Mn, Na
____	2	1 liter plastic	Cool 4° C	F, Cl, SO_4, pH, SC
____	3	250 ml plastic	0.25 ml H_2SO_4	NO_3, TOC
____	4	1 liter glass	1 ml H_3PO_4, 1 g $CuSO_4$	Phenols
____	5	1 liter glass	Cool, 4° C	Pesticides*, Herbicides**
____	6	250 ml glass	Cool, 4° C, Sodium Thiosulfate	Coliform
____	7	1 liter glass	1 ml HNO_3	Gross alpha, gross beta, radium-226, 228
X	(8)	~~1 liter~~ plastic 1L.	~~1 ml HNO_3~~	(TOX)

(Circle parameters for analysis)

Shipping Information

Shipped or delivered to lab by Vicki May
Date 12/19/84 Time 8:30 AM

I hereby certify that to the best of my knowledge ground water samples listed above were obtained in accordance with Marathon Oil Company, Illinois Refining Division's filed sampling and analysis plan and are safely containerized and labeled for delivery to the laboratory.

Signature Victoria May

RECEIVING LABORATORY ETC
Address 1.5.
Attn.

QUADRUPLICATE TESTS REQUIRED FOR:
☒ TOC (TOX)

____ All samples received intact.

____ List samples missing or damaged.

Date Received _____ Time _____

Accepted by _____

Distribution:
white - w/shipment to laboratory
green - w/shipment to laboratory, completed, and returned to Robinson Env. Dept.
~~pink - to Carry Phillips~~
yellow - to Robinson Env. Dept.

* Pesticides = Endrin, Lindane, Methoxychlor, Toxaphene

** Herbicides = 2,4-D and 2,4,5-TP Silvex
Bottle to be capped with aluminum foil or teflon

Project No. _____
Book No. _____ TITLE TOX

Instr. 2	Instr. 3	Instr. 4
c 0.91	method blk 1.56	METHOD BLK 0.93	METHOD BLK 1.91
k 0.96	calib blk 1.07	CALIB BLK 1.22	*CALIB BLK 0.40
std. 6.22 (102.8%)	calib. std 7.09 (109.6)	CALIB STD 4.87 (70%)	*CALIB BLK 0.99
			CALIB STD 4.33 (48.4%)
12	G5513	G 5515	G 5514
E	T	T	T
CALE	B OFF SCALE	B OFF	B OFF
↓	T	T SCALE	T SCALE
	B ↓	B ↓	B ↓
ished	cell flushed	cell flushed	CELL FLUSHED
blk 1.242	method blk 1.427	METHOD BLK 1.80	METHOD BLK 3.09
blk 1.03	calib. blk 1.08	CALIB BLK 1.02	CALIB BLK 1.24
std. 5.62	calib. std. 6.13	CALIB STD 5.91 (90.8%)	* CALIB STD
0	G5509	G 4527	
1 C47 (30.1)	T 29.03 } 468.2	T 2.68 } 14.8	* MACHINE WENT OFF
r .43	B 20.91	B 1.54	SCALE WITH CALIB STD.
292 (31.1)	T 28.55 } 487.7	T 4.17 } 35.6	discontinued use.
1.35	B 23.35	B 1.17	FLUSHED CELL
		T 3.82 } 27.8	
59	G5511	B 1.70	
86 (35)	T 8.74 } 73.3	T 6,22 } 48.6	
1.80	B 1.71	B 1.36	
1.77 (30.5)	T 8.76 } 73.9	CELL FLUSHED	
1.44	B 1.75		
flushed	cell flushed		
l blk 1.08	method blk 1.69		
blk .99	calib. blk 1.33		
b. std 5.87	calib. std 6.02		
ts.			
at next	(cont next		
page)	page)	XMB 1.37	XMB 1.91
		XCB 1.12	XCB .99

To Page No. _____

ssed & Understood by me,	Date	Invented by	Date
1.08	2/6/85	E. Kolen	2/5/85.
B 0.983		Recorded by Paul Ben Smith	

TITLE TOX / Calculations

From Page No.

Instr 1

G4216B
T 2.56 } 14
B 1.20 }
T 2.96 } 23.4
B 1.54 }

G 5519
T 6.77 } 86.5
B 1.73 }
T 6.17 } 53.4
B 1.33 }

CELL FLUSHED

Instr. 2

G5016
T
B
T
B

Sample #4261
T 5.72 } 58
B 3.20 }
T 6.69 } 59.1
B 2.34 }

Sample # G5518
T 3.86 } ~~21.6~~ B.S 23
B 1.42
T 3.71 } ~~H2.9~~ 21.5 B.S
B 1.12 }

CELL FLUSHED

G 5570 - 3.6
G 5516 - 172
G 5519 - 98.8
G 5520 - 128
G 5511 - 33.4
G 5521 - 138
G 4242 - 21.6
G 5518 - 66.2
G 4259 - 17.6
G 4527 - 18.9
G 4261 - 62.5
G 4260 - 46.6
G 9859 - 281

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Ben Smith	2/5/85